

# HIGH PERFORMANCE MULTILAYER OPTICAL COATINGS ON FLEXIBLE SHEET

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## ABSTRACT

Multilayer optical coatings are applied increasingly to transparent polymer substrates. For example, high performance anti-reflective coatings are being applied to display windows on mobile telephones and automotive displays. These developments pose a challenge because the applications require high throughput combined with demanding, but flexible, optical performance with a range of multilayer coating designs. The polymer is often in the form of pre-hard coated sheet suitable for use as an insert in an injection moulding tool.

An optical coating system is described which has been designed specifically to coat flexible polymer sheet using a 0.5m cylindrical drum and up to four 1m linear magnetrons. Throughput is important and 2 drums are used alternately to optimise loading. A hydrophobic topcoat process is included for the easy clean-ability required in many consumer applications.

## INTRODUCTION

Multilayer optical coatings are being applied increasingly to sheets of transparent polymer substrates such as polycarbonate. Applications include anti-reflection (AR) coatings, electrochromic coatings and photovoltaics. In this paper we report on the development of a system that produces uniform coatings on sheets 0.6m x 1.55m on a 0.5m cylindrical drum and utilizing 1m linear magnetrons in a closed field configuration.

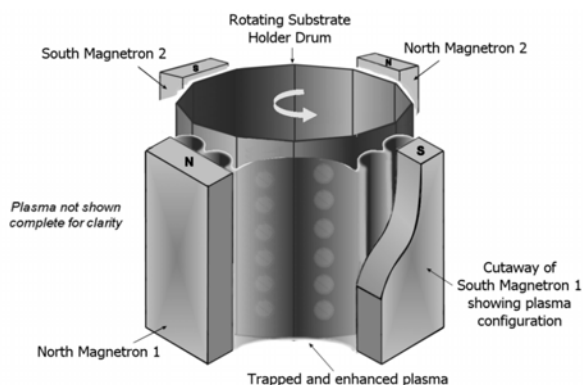
This arrangement allows coatings to be deposited at high rates, typically 0.3nm/sec, while maintaining a temperature below 60C on the polymer surface. The system has 4 magnetron positions to provide flexibility and choice of materials and is also equipped with a thermal evaporation source for the application of a super-hydrophobic top coating.

Throughput is an important issue and for this reason the cylindrical drum is demountable. This allows the drum to be rotated through to the horizontal so that the sheet can be easily fed onto the drum and held under tension. Provision of two drums allows one to be located in the deposition system while a fresh substrate sheet is being applied to the other.

It has been reported previously<sup>1,2</sup> that "closed field" reactive magnetron sputtering produces dense, smooth, spectrally stable metal-oxide optical coating material with refractive indices typically close to that of the bulk material. The thin films also exhibit low stress and this is important if the polymer sheet is to be used subsequently as a label in an injection moulding process<sup>3</sup> with high temperature differentials.

## REACTIVE CLOSED FIELD MAGNETRON (CFM) SPUTTERING

The closed field magnetron sputtering process was developed in 1991 by Teer<sup>4</sup> and has been applied for some time in the field of wear resistant and other tribological coatings<sup>5</sup>. Recently, the necessary control has been developed to cope with process hysteresis with dielectric metal-oxides to enable sub-nanometre thickness precision<sup>6</sup> for application to multilayer optical coatings.



**Figure 1.** The “closed field” magnetron sputtering process does not need a separate ion source. Adjacent magnetrons are made opposite polarity to trap the plasma all the way round the drum. This ensures that all the metal deposited by the magnetrons is converted into metal-oxide.

The closed field process is illustrated schematically in Figure 1. The closed field configuration was developed to increase the ion current density in the plasma by arranging that neighbouring magnetrons are of opposite magnetic polarity. Using this arrangement, the deposition volume in which the substrates are located is surrounded by linking magnetic field lines. This traps the plasma region, prevents losses of ionising electrons, and results in significant plasma enhancement. The closed field system produces enhanced reactivity due to the high ion current density ( $>1\text{mA.cm}^{-2}$ ). The ion energy is determined by the induced Voltage on the substrate carrier which is typically in the range (20-30)eV. This combination of high ion current density and low ion energy produce ideal conditions for growth of optical quality thin films.

The key advantage of the process is that no separate ion source, plasma source or microwave ion source is required. Nor is it necessary to partition the working vacuum chamber into deposition and reaction zones. This simplifies the system, reduces heat load, reduces cost, and improves reliability. It also makes it economical to scale the technology to virtually any batch size.

### THE CLOSED FIELD MAGNETRON (CFM) SPUTTERING SYSTEM FORMAT

The thin film materials presented in this paper were deposited using a CFM850 system from Applied Multilayers Ltd., and shown in Figure 2a. It uses a 550mm diameter vertical-axis drum substrate carrier and up to four 1050mm linear magnetrons. The drum can be

demounted from the coating chamber as shown in Figure 2b and rotated to a horizontal position for application of flexible sheet.

The distance between the magnetron and the segmented drum surface is 100mm. Curved substrates are loaded behind apertures in the segment to ensure a reasonably constant average target to substrate distance.

The system used in these trials was pumped using two 2000 litres/sec BOC Edwards magnetically levitated turbo pumps. A Meissner trap is used for rapid removal of water vapour and reduced pump-down times.

In order to achieve rapid pumpdown from atmosphere the system is pumped by a mechanical booster backed by a two-stage rotary pump (both from BOC Edwards): with this arrangement it is possible to run a complete 4-layer AR coating cycle, with an evaporated hydrophobic topcoat, in about a 30 minutes total cycle time.

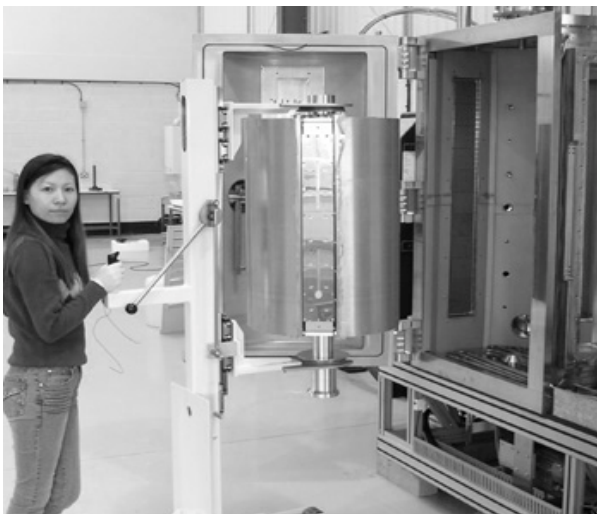
A curved metal plate can be rotated coaxially around the drum to prevent material sputtered from a magnetron arriving on the drum. This shutter is used when conditioning the magnetron targets after exposure to atmosphere.

The coating drum can be removed from the deposition chamber using a manually-operated trolley: this then carries the drum to a semi-automated unit which is used for unloading coated film and then loading new uncoated film onto the drum. This allows two identical drums and trolleys to be used for rapid turn-around, or alternatively a third drum can be used to coat non-film parts such as lenses: this third drum is designed to accept a wide range of different tooling for different substrates.

The deposition chamber, which is water-cooled by a recirculating chiller, is lined with conventional metal shields which can be demounted from the chamber for removal of coating deposits by grit-blasting.



**Figure 2a.** CFM850 coating system from Applied Multilayers Ltd. The system is also fitted with a rotating shutter to expose the substrate to the appropriate target. The shutter is also used to enable targets to be conditioned without exposure to the substrates.



**Figure 2b.** Demountable drum with drum handling tool. A separate tool is used to apply sheet under tension to the drum.

## PROCESS CONTROL

Optical coatings primarily consist of dielectric metal-oxides. Silicon dioxide is almost always chosen as the low refractive index material while the high refractive index material can be chosen from materials such as Zirconia, Niobia, Tantalum, and Titanium. A key advantage of the

closed field process is that the magnetron targets are simple metals. This allows high deposition rates to be achieved using pulsed dc power. The metal is converted to metal-oxide by carefully controlling a reactive plasma.

During reactive oxidation in the closed field process, the oxygen is admitted into the unbalanced magnetron plasma. Target oxidation occurs but is controlled using target Voltage control with feedback to the mass flow of oxygen. Surface charging is overcome by using pulsed dc power (typically >100kHz). Any non-oxidised metal in the film results in absorption. The specific Voltage is optimized for each material to produce non-absorbing films at the highest possible rate. This Voltage corresponds to a particular level of oxidation of the target surface. It is important that this same degree of oxidation is maintained along the full length of the target so that the deposition rate is the same at all points. With careful design of gas manifolds it is possible to obtain +/-1% uniformity over a 0.6m rotating drum.

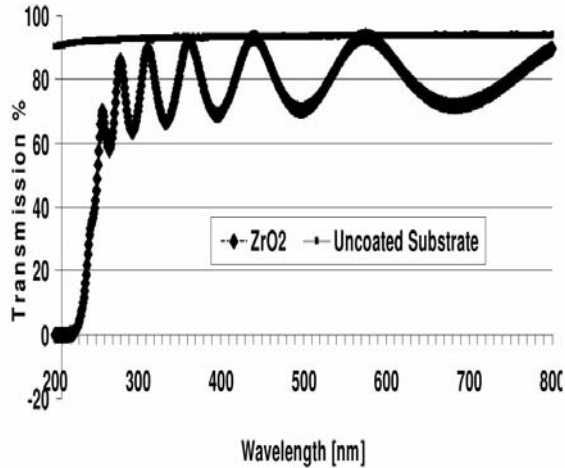
The sputtering rate under these control conditions is constant and thin film thickness is controlled using time only. There is no requirement for quartz crystal monitoring. The times required for each layer are entered into the system computer and these activate the shutter for fine control. This provides thin film thickness accuracy to 1% which is adequate for most applications. For highly demanding applications the system has been designed to accommodate optical monitoring.

Temperature is an important consideration for polymeric substrates. The closed field process does not require additional energy from an auxiliary ion or plasma source and this reduces the heat load to the drum. In the present work the temperature of polycarbonate sheet during a process of typically 15 minutes duration was < 60C while maintaining deposition rates on the drum for SiO<sub>2</sub> and ZrO<sub>2</sub> of 0.3nm/sec.

## OPTICAL MATERIALS

The closed field process is very flexible. It can be used to deposit metals, alloys, metal-oxides, metal-nitrides, metal-oxynitrides and metal-carbides. In this paper, we will focus on the use of metal oxides. The deposition energy, ion current density combined with the stoichiometric control provided by Voltage control in the closed field process provides materials with exceptional optical quality. It has been reported earlier that the materials are dense, specially stable and super-smooth<sup>7</sup>. The materials are also highly transparent and non-absorbing. This is illustrated in the case of a zirconia thin film on a Suprasil glass substrate in Figure 3.

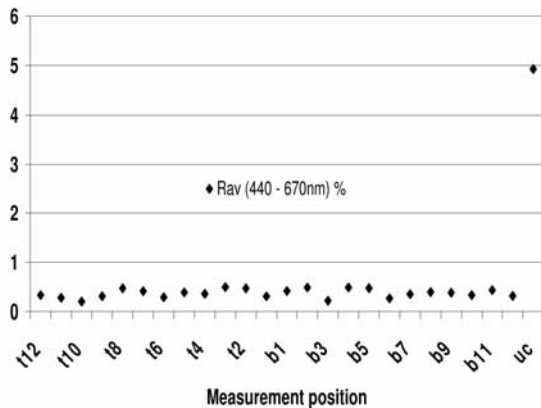
Suprasil is non-absorbing to 200nm. This shows that the zirconia is non-absorbing to 300nm which is the theoretical limit for the bulk material



**Figure 3** The transmission of a niobia thin film on Suprasil showing non-absorption down to the theoretical limit for bulk material.

**ANTI-REFLECTION COATINGS**

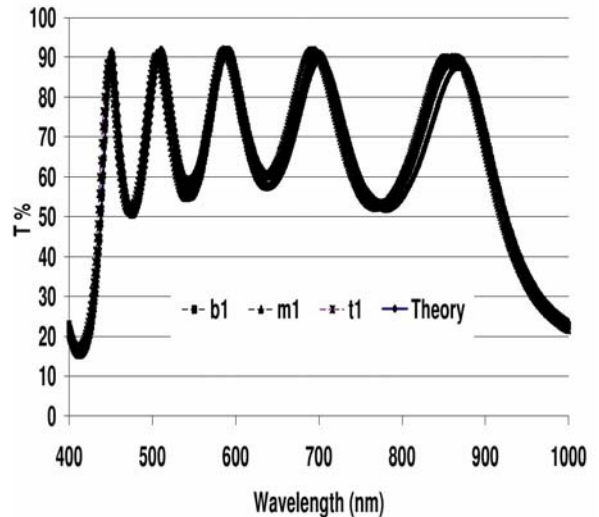
Performance of a low reflectance six layer ZrO<sub>2</sub>/ SiO<sub>2</sub> anti-reflection coating overcoated with a hydrophobic layer is shown in Figure 3. Average reflectance (440 to 670nm) at twenty four positions along sheet length were measured.



**Figure 3** Average reflectance (440 to 670nm) at twenty four positions along sheet width. UC indicates uncoated sheet reflectance

Results indicate average reflectance less than 0.5% measured at twenty four positions along the sheet width.

Ability to deposit more complex multilayers is demonstrated through deposition of a calibration multilayer based on ...

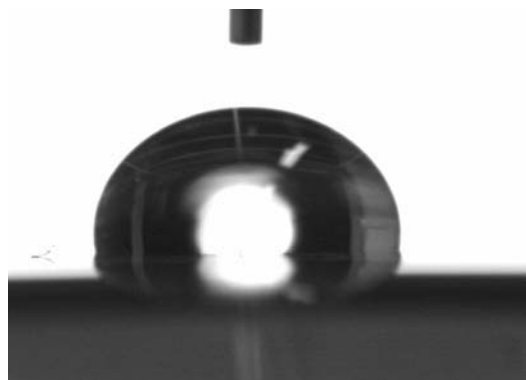


**Figure 4** Spectral transmission of ripple test at three sheet width positions (bottom, mid and top) over five consecutive coating runs

**HYDROPHOBIC COATINGS**

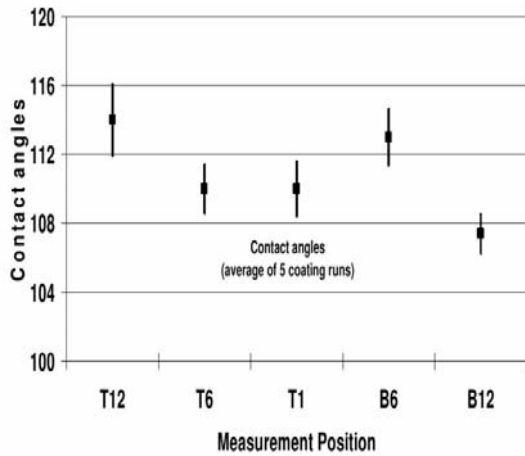
Subsequent to deposition of the all dielectric anti-reflection multilayer a hydrophobic layer is deposited. Hydrophobic layer deposition is achieved by thermal evaporation from boat sources mounted in the front door of the deposition system.

Characterisation of the hydrophobic layer is through measurement of water drop contact angle on the hydrophobic layer surface. An imaged drop is shown in Figure 5.



**Figure 5** Imaged water drop on hydrophobic layer

Figure 6 shows measured contact angle as a function of position on sheet width. Spread in data points is range of contact angles measured at sheet width position over five consecutive coating runs.



**Figure 6** Contact angle at five positions on sheet width and averaged (spread shown) over five consecutive coating runs

## CONCLUSIONS

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